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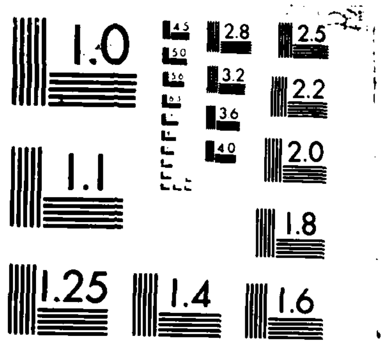

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TECHNICAL REPORT ARCCB-TR-87002

GROOVE ADHESION TEST FOR ELECTRODEPOSITED CHROMIUM

S. K. PAN

E. S. CHEN

JANUARY 1987

US ARMY ARMAMENT RESEARCH, DEVELOPMENT AND ENGINEERING CENTER

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

A quantitative test has been devised to evaluate the adhesion of electrodeposited chromium on steel substrates. The test involves the cutting of parallel grooves across a plated surface using a small metal shaper equipped with a carbide tool. The grooves are cut at a depth just below the interface and shearing stresses are generated which can produce failure of the coating. In general, varying amounts of residual chromium are left on the surface of the

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20. ABSTRACT (CONT'D)

lands depending on the relative cohesive and adhesive bond strengths of the electrodeposited chromium. Energy dispersive x-ray analysis is used to map the distribution of residual chromium and obtain an intensity count. The ratio of intensity count normalized against a reference of 100 percent chromium coverage provides a quantitative measurement of adhesion. The groove adhesion test was found to be equally applicable for the evaluation of both hard chromium and soft chromium deposits.

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ACKNOWLEDGEMENT

The authors wish to express their appreciation to their colleague Leo McNamara for SEM analysis.

INTRODUCTION

Electrodeposited coatings are commonly used to improve the decorative appeal of metal surfaces and to protect them against corrosion and wear. In all such applications, adhesion is a parameter of primary consideration because it determines whether or not a coating will become detached during service. The need to evaluate coating adhesion is apparent; however, the difficulty is that very often, individual adhesion tests must be developed to meet a specific need.

A large number of methods, both qualitative and quantitative, are used to measure adhesion. Descriptive accounts of these methods are available in ASTM standards (ref 1), journal articles, and critical reviews (refs 2-6). In general, the selection of a suitable adhesion test method depends to a large extent on whether thin films or thick coatings are to be tested and whether the coatings are hard and brittle or soft and ductile. For hard, thin films, the scratch test has been found to be capable of providing a quantitative measure of adhesive strength. The basic model was derived on the assumption that the interface is subjected to a shear force produced by the motion of a loaded stylus. Adhesion strength is identified with the critical load at which the coating spalls.

Generally, thick, brittle deposits are not amenable to the scratch test unless a chisel or other sharp instrument is used in conjunction to expose the coating/substrate interface (ref 1). The groove test (ref 7) was developed using features associated with the hybrid scratch test. In this case, coating failure is produced by the grooving action of a shaper cutting tool. As a

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References are listed at the end of this report.

qualitative tool, optical microscopy is used to examine the exposed interfacial area of a failed region; however, quantified adhesion strength can also be obtained when analytical instrumentation is applied to determine the degree of exposed interfacial area. This report describes the use of energy dispersive x-ray analysis (EDAX) with the groove test. Quantitative adhesion measurements of thick chromium deposition on steel are presented.

EXPERIMENTAL PROCEDURES

The groove test was developed specifically to evaluate the adhesion of chromium electrodeposits on steel substrates. The test procedure involves (a) cutting parallel grooves across the plated surface to induce coating failure, and (b) analyzing the fractured surface with EDAX to determine the relative concentrations of residual chromium and exposed steel. The ratio of intensitites of residual chromium to a reference of 100 percent chromium provides a quantitative measure of adhesion strength.

Grooving

The procedures for conducting the groove test have been reported in earlier publications (refs 7-8). For this test, specimen geometry is not crucial provided the specimen can be held securely for a cutting tool to traverse evenly across the plated surface. In this study, a small metal shaper equipped with a carbide tool is used to cut parallel grooves on the surface of chromium plated steel samples as illustrated in Figure 1. Since shear stresses necessary to fracture the coating are generated by the grooving tool, the correct geometry of the tool tip is critical to achieve optimum stresses consistently. From tests with different tool configurations, the best design was found to be a tip machined with a top rake angle of 15 degrees and an included angle of 90 degrees, Figure 2. Other test parameters remain unchanged, i.e., deposit thickness > 75 μm, grooving depth at 0.3 mm below the coating/substrate interface, and a groove separation of 1.0 mm.

Evaluation of Fracture Surface

When the chromium coating is delaminated by the grooving process, fracture takes place either at the chromium/steel interface or within one of the metals depending on the relative cohesive and adhesive bond strengths. Because chromium deposits contain microcracks and are highly stressed, it is reasonable to assume that delamination will occur in the chromium deposit and/or chromium/steel interface. Therefore, the amount of chromium remaining on the land surfaces (area between grooves) provides a measure of adhesion of chromium to steel.

Energy dispersive x-ray mapping is employed to reveal the chromium distribution on the fractured surface, Figure 3. The chromium x-ray intensity is proportional to the amount of residual chromium on the surface. The total intensity is counted for a given period of time, e.g. 100 seconds, and normalized against a reference intensity of a 100 percent chromium coverage. The degree of chromium adhesion to the steel substrate is calculated in terms of percent of coverage.

An ETEC Autoscan-U1 scanning electron microscope furnished with an energy dispersive analyzer, KEVE-7000, was used in the analysis. The grooved specimen was cleaned to remove the loose fragments and particles of the fractured chromium in preparation for scanning electron microscopy. Test specimens were tilted 30 degrees from a horizontal position toward the electron detector to obtain the overall surface image. The specimens were subsequently tilted to 40 degrees and the condenser lens current in the microscope set at 1.5 Amp for the chromium x-ray mapping and intensity counting over a period of 100 seconds. The electron beam was generated at 20,000 volts.

An identical specimen grooved by precision grinding to insure perfect chromium coverage on the lands was used as a reference of 100 percent chromium adhesion. The reference specimen was tested under the same conditions as the fractured specimens to obtain intensity counting for normalization.

RESULTS AND DISCUSSION

The groove adhesion test was used to sample a large group of chromium plated steel specimens. The tests were divided into two categories. Specimens in the first category were plated using pretreatment processes known to produce different degrees of adhesion. Four degrees of adhesion were produced and classified as excellent, good, fair, and poor. Table I shows a comparison of the groove adhesion test data with the known values. Figures 4 and 5 show typical chromium x-ray mapping of specimens with good adhesion and poor adhesion. Figure 6 illustrates the distribution of perfect chromium coverage on the reference specimen.

From these results, it is clear that the groove adhesion test has the sensitivity to differentiate the degree of chromium coating adhesion to the steel substrate with respect to both the strength and the uniformity of interfacial bonding. Because the chromium x-ray can be produced by both the incident electron beam and the iron x-ray, these measurements can theoretically include some error contributed from the fluorescent x-ray induced by the excitation of iron x-ray in the steel substrate. Since the chromium x-ray intensity counting and mapping are based on the chromium x-ray produced only by the incident electron beam striking the residual chromium on the fractured surface, it is necessary to assure that the iron x-ray bordering chromium does not contribute to an enhancement of x-ray intensity. Tests were conducted showing that this was indeed the case. The effect on the counting of chromium intensity was negligible and no effect was observed on the chromium x-ray mapping.

TABLE I. GROOVE ADHESION TEST DATA ON VARIOUS BONDING OF ELECTRODEPOSITED CHROMIUM ON STEEL

Specimen No.	Substate Surface Preparation	Coating Thickness (μ)	Groove Adhesion Test	
			Chromium (%)	Chromium Distribution
CR1ML	Reference	25	100	Completely Covered (Fig. 6)
HC4P1	Excellent	168	73	Uniform (Fig. 4)
HC4P3	Good	177	66	Spotty
HC4P7	Fair	147	42	Spotty
HC4P5	Poor	147	37	Area (Fig. 5)

The fracturing of coatings by grooving is a complicated process. The mechanism of coating delamination is beyond the scope of this study. However, the parameters of importance to the grooving process, such as tool geometry, groove speed, groove depth, and groove separation, which determine the sensitivity and reproducibility of the method, have been optimized experimentally and reported previously (refs 7-8). A double-tip tool has been designed for future testing. The design is based on the depth of cut and the separation of the groove for producing a land of constant width and acted on by a uniform shear stress. It is expected that this refinement will improve the consistency of test conditions.

In order to compare the results of the groove adhesion test to the proof firing test, the second category of test specimens was prepared from the chromium plated extension rings of the 120 mm M256 gun tubes. Chromium coatings were rated by firing tests and grouped into good, medium, and poor. Table II lists both test data, the proof firing test (after 13 rounds) showing chromium loss area in (mm^2), and the groove adhesion test showing residual chromium on land area in (%). There is good agreement between the two tests for the specimens rated in the groups of good and medium. However, in the group of poor the chromium coatings have much better adhesion. It is quite reasonable to assume that the high chromium loss from those good adhesion coatings is of cohesive fracture, i.e., instead of chromium breaking off from the interface of the coating and steel, it flaked off the coating partially.

TABLE II. EVALUATION OF ADHESION OF CHROMIUM COATING AND COMPARISON OF EROSION DATA

Specimen Ring No.	Proof Firing Test		Cr-Thickness	Groove Adhesion Test	
	Rating	Cr Loss (mm^2)	(μ)	Cr (%)	Cr Distribution
212	Good	10	146	81.8	Uniform
215		0	146	57.2	Uniform
223		20	113	87.8	Uniform
239	Medium	400	130	64.4	Spotty
241		500	135	68.4	Spotty
248		200	136	62.8	Spotty
270	Poor	3835	147	95.4	Uniform
309		4187	163	85.8	Uniform
291		5827	178	95.8	Uniform

CONCLUSIONS

The above results show that the chromium loss data include both the adhesive and cohesive fracture of chromium coating. SEM/EDAX data in the groove adhesive test reveals only the adhesive fracture of the chromium coating. Therefore, evaluation of the grooving chips to determine the combination of cohesive and adhesive fracture should be continued in groove adhesion tests. The complete groove test will provide the method of chromium plating quality evaluation, including coating adhesion as well as the combination of the cohesive and adhesive bonding strength of chromium coating on gun steel.

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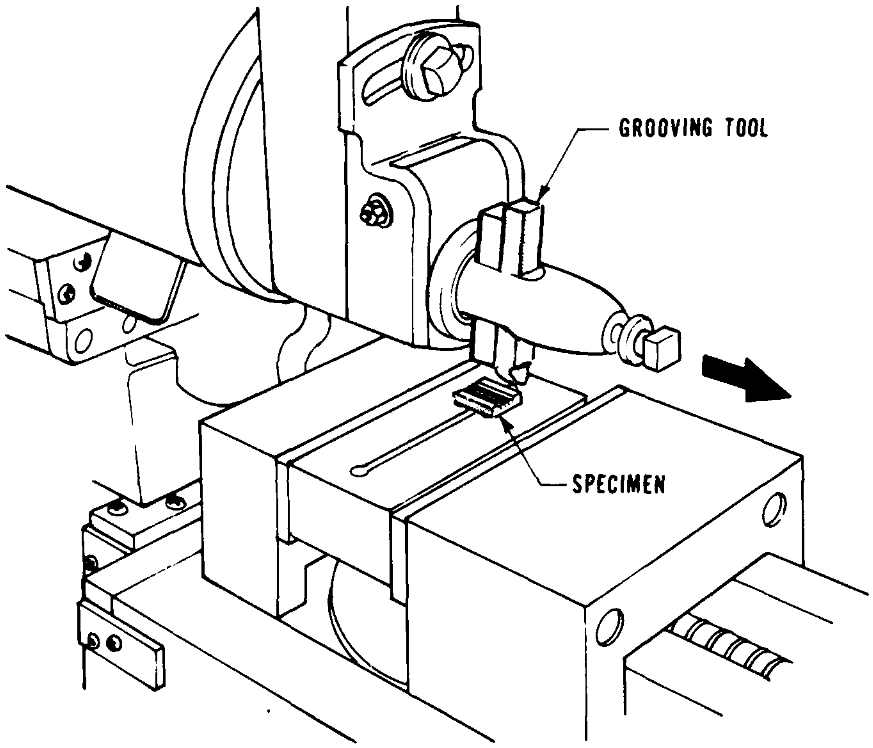

Figure 1. Schematic diagram of the groove adhesion test apparatus.

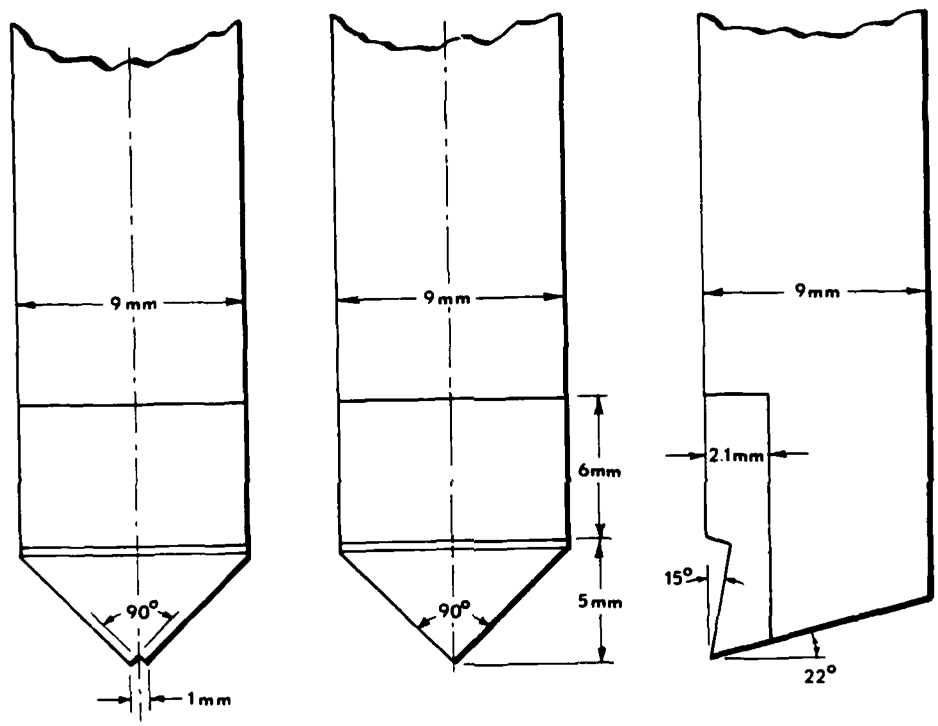

Figure 2. Geometry of grooving tool.

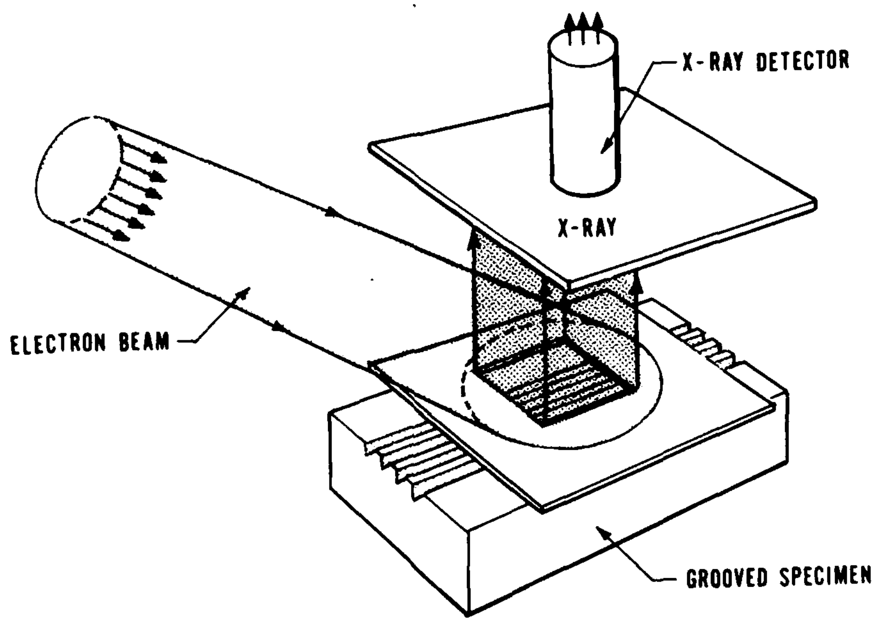

Figure 3. Energy dispersive x-ray analysis set up for the evaluation of groove adhesion test.

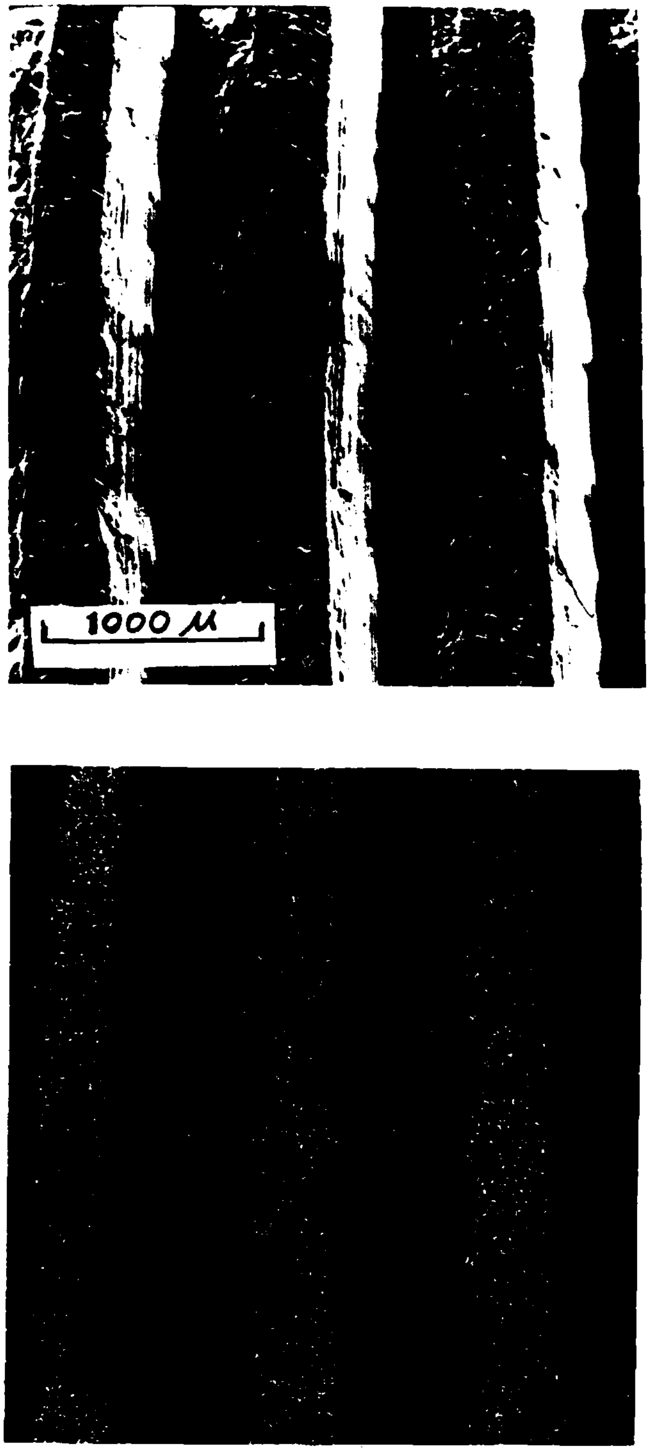

Figure 4. Photograph and chromium x-ray mapping of electrodeposited chromium showing good adhesion.

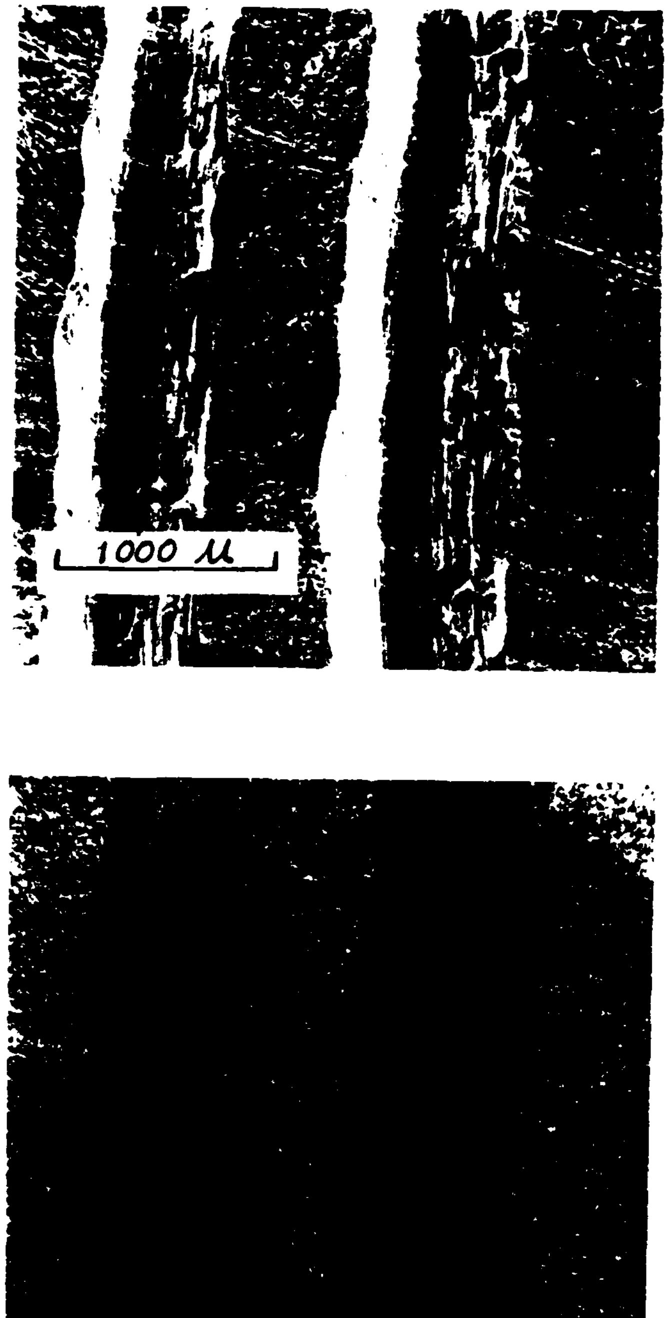

Figure 5. Photograph and chromium x-ray mapping of electrodeposited chromium showing poor adhesion.

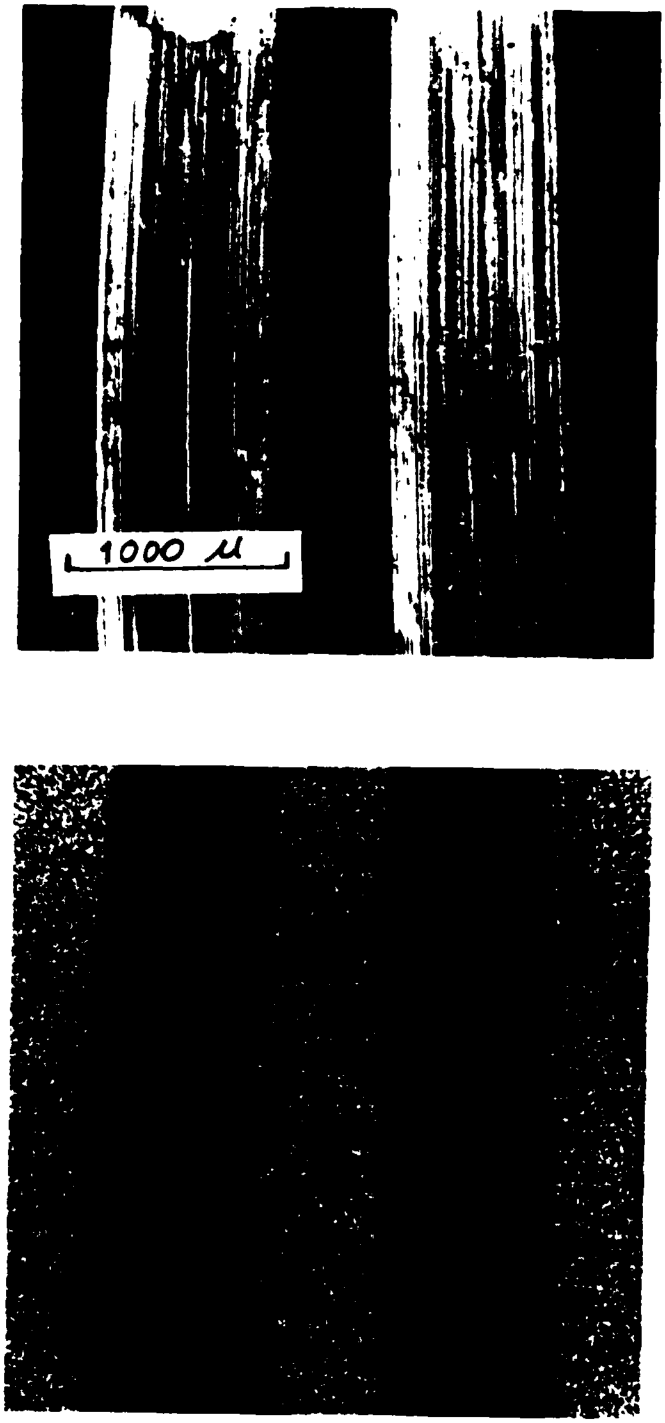

Figure 6. Photograph and chromium x-ray mapping of reference specimen.

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